

Exhibit 7



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MAY 27 1987
GROUP 130

CASE 3528

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In the Application of

DAVID C. HECKERT :

Serial No. 860,607 :

Filed May 7, 1986 :

Group Art Unit 132

Examiner C. Paden

FRUIT JUICE BEVERAGES AND

JUICE CONCENTRATES NUTRI-

TIONALLY SUPPLEMENTED WITH CALCIUM

W. P. D.
W. P. D.
6-1-82

DECLARATION OF TIMOTHY W. DAKE PURSUANT TO 37 CFR 1.132

I, Timothy W. Dake, declare as follows:

1. I am familiar with the subject matter of the present invention disclosed and claimed in the above application.
2. I received a B.S. degree in Chemical Engineering from the University of Toledo in 1972, and an M.S. degree in Chemical Engineering from the University of Toledo in 1973. I have been employed at The Procter & Gamble Company, the assignee of the above application, since September of 1973, and have been involved in its beverage program since August of 1977.
3. I was present at the interview of the above application which took place on March 2, 1987. At this interview, single-strength samples of orange and grapefruit juice beverages of the present invention, similar to those shown in photograph 1 attached to this Declaration, were offered to the Examiners. The juice content (single-strength basis), calcium content, total acid level (combined citric and malic acid) and weight ratio of citric to malic acid of these beverages (calculated) were as follows:

	<u>Orange</u>	<u>Grapefruit</u>
Juice Content	60%	60%
Calcium Content	0.137%	0.124%
Total Acid Level	1.42%	1.99%
Ratio Citric:Malic	72:28	81:19

4. The samples of orange and grapefruit juice beverages of paragraph 3 were prepared at my direction by combining distilled water with the

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respective calcium-supplemented orange or grapefruit juice concentrate of the present invention in a 3 to 1 volume ratio. The calcium content, total acid level (combined citric and malic acid) and weight ratio of citric to malic acid of these concentrates (calculated) were as follows:

	<u>Orange</u>	<u>Grapefruit</u>
Calcium Content	0.48%	0.45%
Total Acid Level	4.95%	7.16%
Ratio Citric:Malic	72:28	81:19

5. The orange and grapefruit juice concentrates of paragraph 4 were prepared according to the premix method of the present invention. In this method, a premix solution of solubilized calcium was initially prepared from the following ingredients:

<u>Ingredient</u>	<u>Orange</u>	<u>Grapefruit</u>
Calcium carbonate	3.03%	2.82%
Citric Acid (anhydrous)	3.88%	2.33%
Malic Acid	2.59%	2.91%
Liquid Sucrose (67.5° Brix)	65.8%	55.85%
Water	Balance	Balance

Calcium Content	1.21%	1.13%
Total Acid Level	6.47%	5.24%
Ratio Citric:Malic	60:40	44:56

The above ingredients were combined as follows: Citric and malic acid were added to the water and mixed in a blend tank until dissolved. Calcium carbonate was added to this acid solution and mixed until carbon dioxide evolution ceased. Liquid sucrose was then added and mixed to provide the premix solution.

The orange and grapefruit juice concentrates were obtained by adding and mixing together the following components in a blend tank:

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<u>Component</u>	<u>Orange</u>	<u>Grapefruit</u>
Premix Solution	37.67%	37.67%
Concentrated Juice	38.06%	34.40%
Aqueous Essences	6.8%	-
Citrus Oils	0.02%	0.026%
Citrus Pulp	5.01%	5.31%
Color	0.006%	-
Vitamins	0.076%	0.06%
Water	Balance	Balance

6. At the March 2, 1987 interview, I also conducted demonstrations to show the problems caused by direct addition of calcium hydroxide and calcium carbonate to orange juice or to orange juice concentrate. The orange juice used in these demonstrations was prepared at my direction by diluting Citrus Hill Select frozen orange juice concentrate with distilled water in a 1 to 3 volume ratio. The orange juice concentrate used in these demonstrations was Citrus Hill Select frozen orange juice concentrate (juice solids content of 42° Brix) which had been thawed. These demonstrations are described in further detail in paragraphs 7 and 8 of this Declaration.

7. I conducted two demonstrations involving direct addition of calcium hydroxide to orange juice. These demonstrations are believed to be representative of what occurs during the direct addition of calcium hydroxide to orange juice or orange juice concentrate in a large blend tank typically used in commercial citrus juice operations. These large blend tanks mix the ingredients in such a way that fairly high, localized concentrations of calcium hydroxide can result in the juice or juice concentrate.

In the first demonstration, I added 1.1 grams of calcium hydroxide to 250 grams of orange juice. (If completely dissolved, this would yield a calcium content in the juice of 0.24%.) I found it very difficult to dissolve the calcium hydroxide in the juice, even with vigorous hand stirring. Photograph 2 attached to this Declaration, which shows orange juice samples before and after calcium hydroxide addition, is representative of what occurred during this first demonstration at the interview.

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(Photograph 2 was taken approximately 10 minutes after addition of the calcium hydroxide to the juice.) As can be seen, the color of the orange juice significantly darkened after calcium hydroxide addition. Although not shown in photograph 2, the color of the juice actually became green in color at the interview. While not occurring at the interview, the juice can also slowly develop a fishy, amine odor.

In the second demonstration, I added 1.1 grams of calcium hydroxide to 125 grams of orange juice. (If completely dissolved, this would provide a calcium content of 0.48% in the juice.) Like the first demonstration, I found it very difficult to dissolve the calcium hydroxide in the juice. Photograph 3 attached to this Declaration, which shows orange juice samples before and after calcium hydroxide addition, is representative of what occurred during this second demonstration at the interview. (Photograph 3 was taken approximately 10 minutes after calcium hydroxide addition.) As can be seen, the color of the juice darkened after calcium hydroxide addition. Also, a gel formed which separated out. It is believed that this gel is the result of pectin in the juice which has been demethoxylated by the hydroxide and then reacted with the calcium. As occurred at the interview, the juice quickly developed a fishy, amine odor. This amine odor is believed to be due to the breakdown of proteins and amino acids present in the juice.

8. The demonstration of direct addition of calcium carbonate to orange juice concentrate is representative of what would occur in a blend tank typically used in commercial citrus juice operations. In this demonstration, I added a mixture of calcium carbonate (1.06 gram), citric acid (0.36 gram), and malic acid (0.98 gram) to 100 grams of orange juice concentrate. (If completely dissolved, this would provide a total calcium content of 0.46%, a total acid level (combined citric and malic acid) of 5.54% and a weight ratio of citric to malic acid of 70:30 in the concentrate.) I found it difficult to completely dissolve this mixture in the orange juice concentrate, even with vigorous hand stirring.

Photograph 4 attached to this Declaration, which shows orange juice concentrate samples before and after the addition of the mixture of calcium carbonate, citric acid, and malic acid, is representative of what occurred during this demonstration at the interview. (Photograph 4 was taken approximately 5 minutes after addition of the mixture.) As can be seen, the orange juice concentrate foamed considerably after addition of

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this mixture due to carbon dioxide evolution as the result of the reaction of calcium carbonate with the acids. This foaming would make pumping and further processing of the concentrate difficult.

9. At the March 2, 1987 interview, a sample of single-strength orange juice to which calcium chloride had been added was offered to the Examiners. This sample was prepared at my direction by adding 1.1 gram of calcium chloride (dihydrate form) to 250 grams of single-strength orange juice which had been prepared by diluting Citrus Hill Select frozen orange juice concentrate with tap water in a 1 to 3 volume ratio. This sample contained 0.13% calcium and 0.21% chloride. As I remember at the interview, the Examiners, after tasting this sample, noticed the salty note imparted by the chloride.
10. At the March 2, 1987 interview, samples representative of products prepared according to Kaji et al were also shown to the Examiners. Photograph 5 is believed to be representative of the products shown, with and without the addition of vinegar. (Photograph 5 was taken approximately 30 days after preparation of the sample with vinegar and approximately 17 days after preparation of the sample without vinegar.) As can be seen in photograph 5, the sample with vinegar has a light brown solid at the bottom of uncertain composition, but which is believed to contain calcium citrate. As also shown in photograph 5, the sample without vinegar contains large amounts of white solid at the bottom which was identified at the interview as being calcium citrate.
11. The Kaji et al samples of Paragraph 10 were prepared from the following ingredients:

<u>Ingredient</u>	<u>Wt. %</u>
Calcium Hydroxide	1.22
Citric Acid (monohydrate)	0.771
Malic Acid	0.779
Lactic Acid	0.826
Dextrose*	2
Sucrose*	1
Orange Juice	3
Lemon Juice	1
Water	89.4

*Replaces fruit sugar and invert sugar of Kaji et al

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The above ingredients were combined as follows: Citric, malic and lactic acid (50% solution) were dissolved in distilled water. Calcium hydroxide was added to this acid solution and mixed in. Dextrose and sucrose were then added and mixed in. Single-strength orange juice (prepared from Citrus Hill Select frozen orange juice concentrate diluted with distilled water in a 3 to 1 volume ratio), and lemon juice (Minute Maid 100% pure lemon juice) were then added and mixed in. In the case of the sample without vinegar, this solution was put into 16 ounce bottles and then capped. This sample without vinegar had a calcium content of 0.66% and a pH of 4.8. In the case of the sample with vinegar, apple cider vinegar was added to this solution (308 grams per 1,000 grams of sample prepared) to adjust the pH to 4.0, which is the preferred procedure in Kaji et al. This vinegar-containing solution was put into 16 ounce bottles and then capped. The sample with vinegar had a calcium content of 0.50%. The amount of vinegar added is so high as to make this sample virtually unpalatable.

12. At my direction, four calcium-fortified orange juice samples (A, B, C and D) having the following levels of added calcium, added citric and malic acid, added chloride, % added calcium from calcium chloride, and pH were paneled:

	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
Added calcium (%)	0.12	0.12	0.12	0.12
Added citric acid (%)	0.017	0.013	0.009	0.005
Added malic acid (%)	0.327	0.250	0.173	0.096
Added chloride (%)	0	0.043	0.086	0.129
% Ca from chloride	0	20	40	60
pH	4.04	3.96	3.88	3.79

Twenty-six untrained panelists who typically panel beverage products were used. Each panelist rated all four samples. Each sample was rated on a scale of 1 to 9 for sweetness, sourness, bitterness, salty/brackish taste, and taste quality, using the form identified as "Taste Test" attached to this Declaration. The results of this paneling are shown in the following table:

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	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
Sweetness	4.23	4.27	4.27	4.19
Sourness	4.65	4.38	4.00	3.65
Bitterness	2.58	2.77	2.46	2.65
Salty/brackish	2.15	2.62*	3.31**	4.96**
Taste quality	4.77	4.69	4.15	2.88

*Not statistically significant difference relative to sample A at a 95% confidence level.

**Statistically significant difference relative to sample A at a 95% confidence level.

13. The calcium-fortified orange juice samples paneled in paragraph 12 were prepared from the following ingredients:

	<u>A</u>	<u>B</u>	<u>C</u>	<u>D</u>
<u>Ingredients</u>				
Citric acid (%)	0.0172	0.013	0.009	0.005
Malic acid (%)	0.327	0.250	0.173	0.096
Calcium hydroxide (%)	0.224	0.179	0.134	0.090
Calcium chloride dihydrate (%)	0	0.089	0.178	0.267
Orange juice concentrate (%)	28.23	28.23	28.23	28.23
Water (%)	71.20	71.24	71.28	71.31

The above ingredients were combined as follows: citric and malic acid were added to the water and dissolved. Calcium hydroxide was then added to this acid solution and mixed in. For samples B, C and D, calcium chloride dihydrate was then added and mixed in. Finally, orange juice concentrate (Citrus Hill Select frozen orange juice concentrate) was added and mixed in to provide the orange juice sample.

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I further declare that all statements made of my own knowledge are true and that all statements made on information and belief are true; and further that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful, false statements may jeopardize the validity of the above application or any patents issuing therefrom.

Timothy W. Dake
Timothy W. Dake

May 7, 1987
Date

3528a

TASTE TEST

No: _____

EMPLOYEE ID#: _____

NAME: _____

TASTE ORDER: _____

DATE: _____

LOCATION: _____

WE WOULD LIKE YOU TO EVALUATE THE FOLLOWING FOUR ORANGE JUICE PRODUCTS FOR SWEETNESS, SOURNESS, BITTERNESS, SALTY/BRACKISH TASTE AND TASTE QUALITY.

SWEETNESS

1	2	3	4	5	6	7	8	9
NONE		SLIGHT		MODER.		HIGH		V. HIGH

SOURNESS

1	2	3	4	5	6	7	8	9
NONE		SLIGHT		MODER.		HIGH		V. HIGH

BITTERNESS

1	2	3	4	5	6	7	8	9
NONE		SLIGHT		MODER.		HIGH		V. HIGH

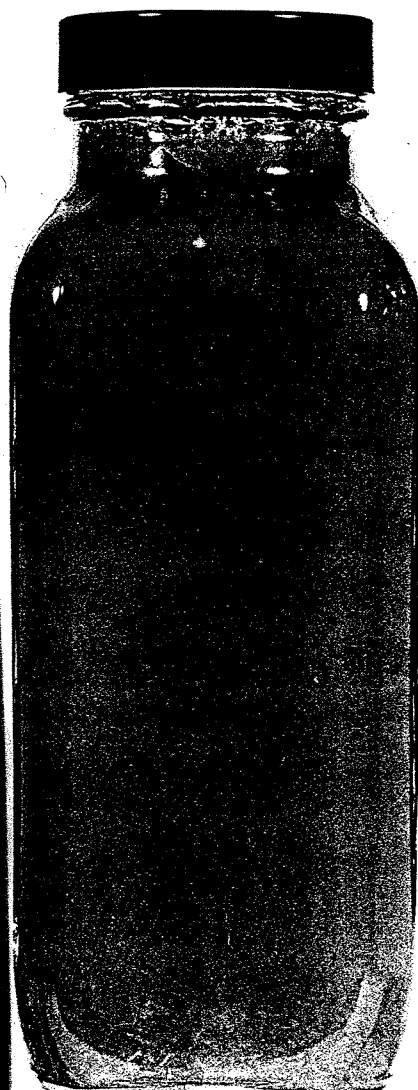
SALTY/BRACKISH TASTE

1	2	3	4	5	6	7	8	9
NONE		SLIGHT		MODER.		HIGH		V. HIGH

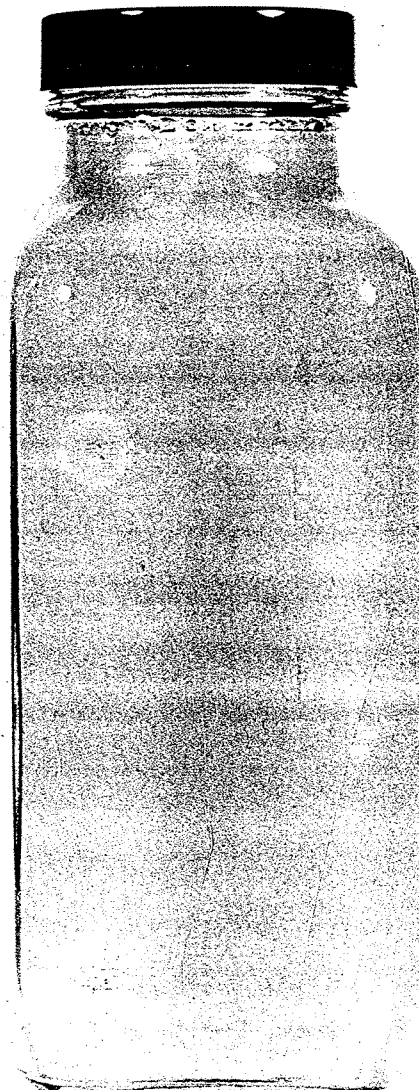
TASTE QUALITY

1	2	3	4	5	6	7	8	9
BAD		POOR		ACCEPT- ABLE		V. GOOD		EXCELL- ENT

THANKS FOR YOUR COOPERATION

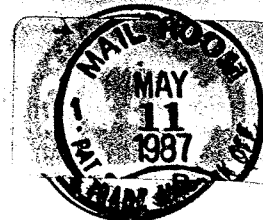


PRESENT INVENTION
60% Orange Juice



PRESENT INVENTION
60% Grapefruit Juice

1087A206-2-K



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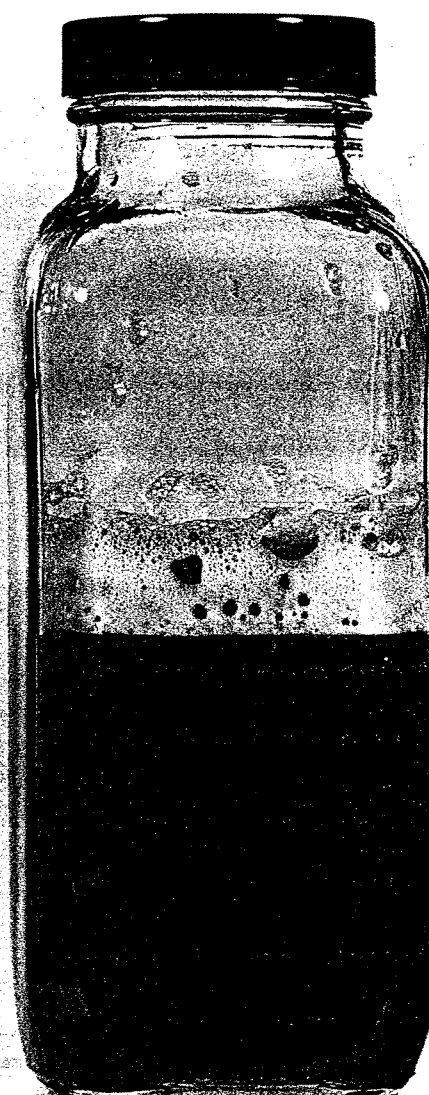
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Orange Juice
before $\text{Ca}(\text{OH})_2$ addition



Orange Juice
after $\text{Ca}(\text{OH})_2$ addition

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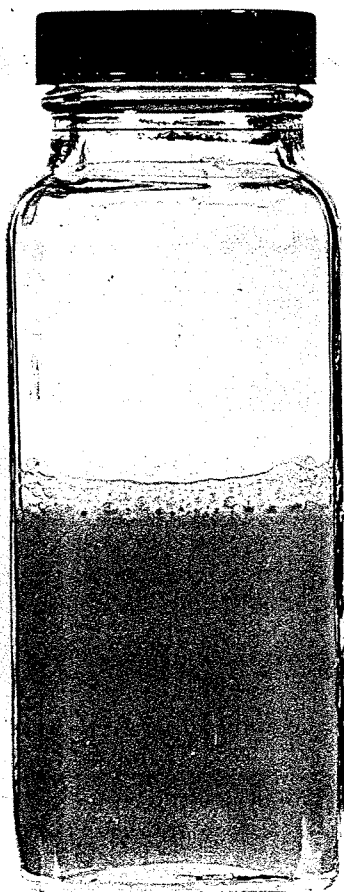
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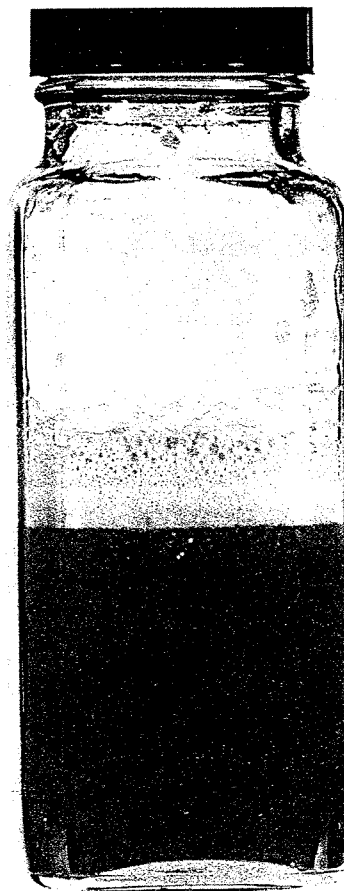
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EXHIBIT 130
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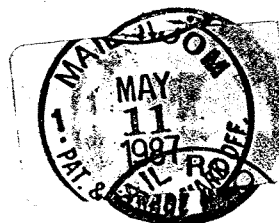
Orange Juice
before $\text{Ca}(\text{OH})_2$ addition

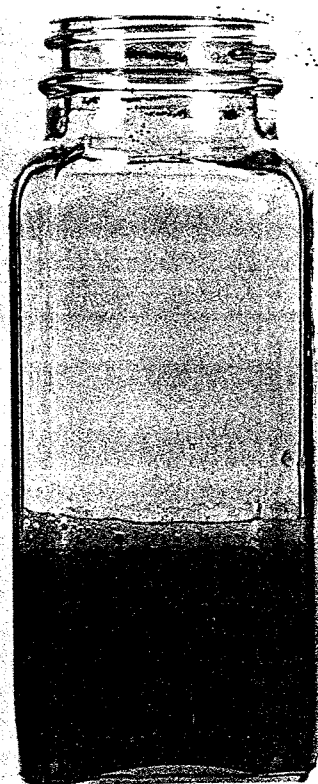


Orange Juice
after $\text{Ca}(\text{OH})_2$ addition

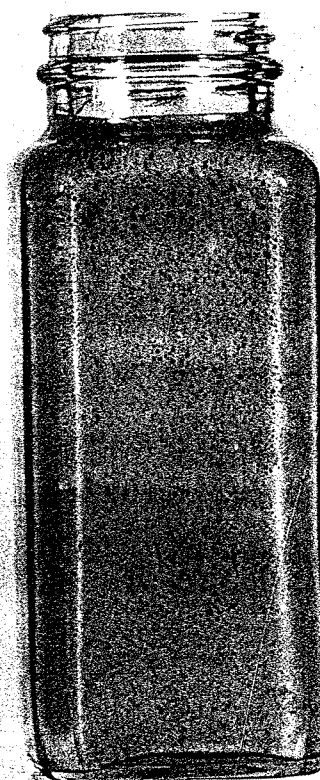
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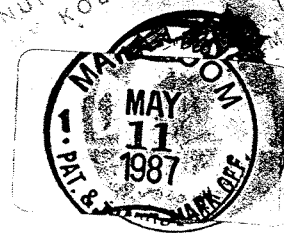


Orange Juice Concentrate
before CaCO_3 : Citric: Malic addition



Orange Juice Concentrate
after CaCO_3 : Citric: Malic addition

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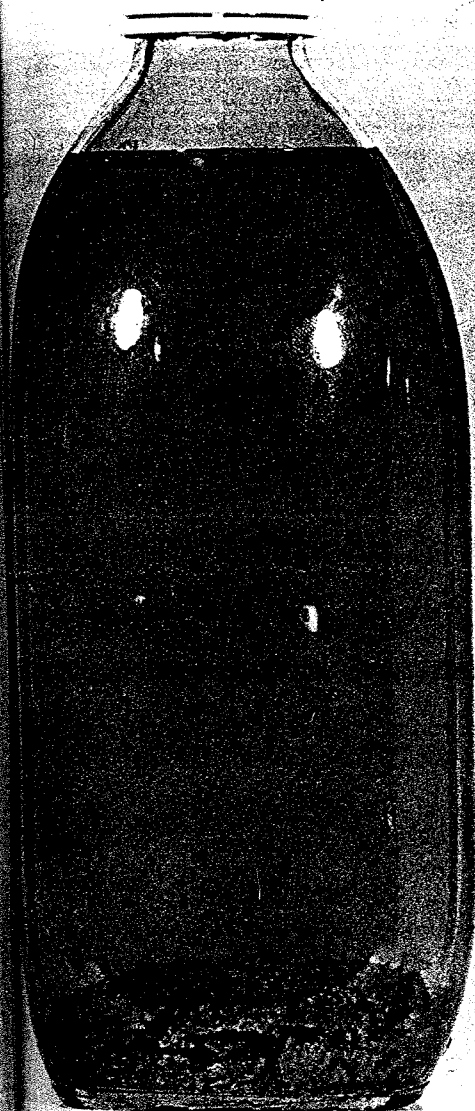


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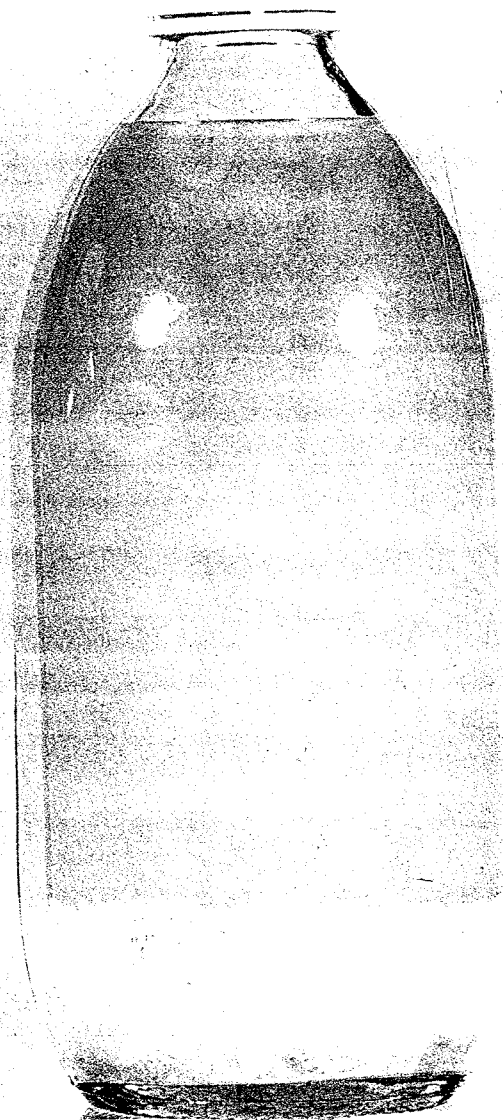
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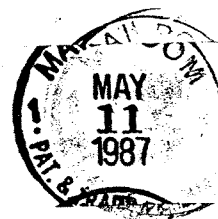


**Kaji
with vinegar**



**Kaji
without vinegar**

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